

# Standard Test Methods for Accumulated Deposition in a Steam Generator Tube<sup>1</sup>

This standard is issued under the fixed designation D 3483; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last approval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover determination of the weight per unit area of waterside deposits on heat-transfer surfaces of steam generator tubes. Two test methods are given as follows:

Test Method A—Mechanical Removal	Sections 6 to 11
Test Method B—Solvent Removal	12 to 18

1.2 Neither test method is normally applicable to fire-tube boilers.

1.3 A comparison of the results obtainable with the two test methods is shown in Fig. 1.

1.4 A scope section is provided in each test method. It is the responsibility of the analyst to determine the acceptability of these test methods for each situation.

1.5 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

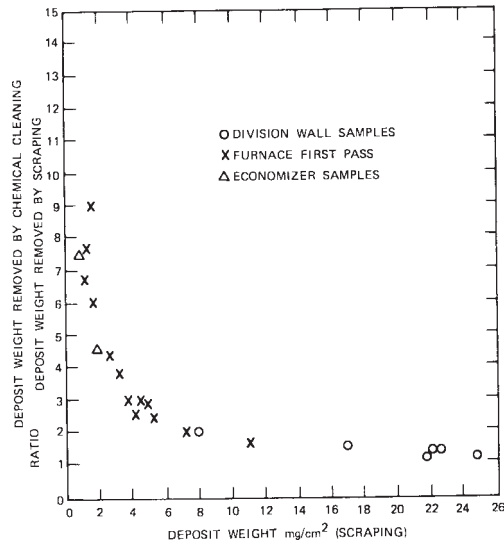
- 2.1 *ASTM Standards:*
  - D 887 Practices for Sampling Water-Formed Deposits<sup>2</sup>
  - D 1129 Terminology Relating to Water<sup>3</sup>
  - D 1193 Specification for Reagent Water<sup>3</sup>

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1129.

## 4. Significance and Use

4.1 The weight per unit area measurement is an indication of the relative cleanliness or dirtiness of the boiler; therefore, it is important that a tube sample be selected that represents near maximum deposition.



NOTE 1—Data for graph were received from Babcock-Hitachi. Methods used were not identical to those described herein.

FIG. 1 Comparison Deposit Weights Determined by Scraping and Acid Cleaning

## 5. Sampling

5.1 Select a tube section likely to have the heaviest deposit. Experience has shown that deposit accumulation is usually heaviest on tube surfaces that receive the highest heat transfer. Representative areas of especially high absorption are:

5.1.1 The center of the division wall at the top burner elevation in a boiler with a division panel wall where firing occurs on opposite sides.

5.1.2 The side wall near the top burner elevation, at about 1/3 furnace depth from the burner wall, in a boiler without a division wall.

5.1.3 Other high heat absorption areas in a more complex boiler design as delineated by the boiler manufacture.

5.2 Areas in the boiler where impaired circulation is suspected may also be sampled.

5.3 After selecting the boiler tube to be sampled, provide suitable identification, showing location in the boiler, the direction of flow, and the hot and shielded sides in accordance with Practices D 887.

<sup>1</sup> These test methods are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of Subcommittee D19.03 on Sampling of Water and Water-Formed Deposits, Surveillance of Water, and Flow Measurement of Water.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.02.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 11.01.

5.3.1 Remove a sufficient portion of the tube to contain a selected 600-mm (24-in.) section, allowing at least 300 mm (12 in.) more on each side of the sample if a cutting torch is used.

NOTE 1—No oil or water is to be used in any mechanical cutting operation.

5.3.2 Separate a selected 600-mm (24-in.) section by careful application of an anchored pipe vise and a tube cutter.

## TEST METHOD A—MECHANICAL REMOVAL

### 6. Scope

6.1 The mechanical removal test method is preferred when deposition is comparatively heavy and the deposits are relatively easy to dislodge. The deposit, so removed, may serve as the sample for determining the composition of the material.

### 7. Summary of Test Method

7.1 A section of the most heavily fouled portion of the sampled tube is selected on a visual basis. After dividing the tube, the water-formed deposit is removed mechanically from a measured area. The weight of the dry material is reported as milligrams of deposit per square millimetre of boiler tube surface.

### 8. Apparatus

8.1 *Cutting Tool or Torch*, removing a suitable portion of boiler tube and a *vise* for crimping.

NOTE 2—Lightly crimping the sample tube in a vise may be effective in removal of very brittle deposits. However, any physical change that the tube specimen is subjected to may effect any subsequent metallographic examination.

8.2 *Tube Cutter*.

8.3 *Tube End Sealers*, to protect the sample if the determination is to be made elsewhere than on the site.

8.4 *Milling Machine (Preferred) or Band Saw*, to separate the fireside half of the tube from the shielded half by longitudinal sectioning (dry cut).

8.5 *Magnet*, to remove metal chips from the deposited material, especially if a band saw is used.

8.6 *Scraping Tool*, for removing the less adherent deposits (like a scalpel or a heavy screwdriver, the end of which has been thinned).

8.7 *Vise*, for removal of brittle deposits.

8.8 *Vibrating Tool*, to remove more adherent deposits (a small head should be available for use within pits).

8.9 *Oven*, for drying the deposits.

8.10 *Analytical Balance*.

### 9. Procedure

9.1 Take the 600-mm (24-in.) tube section obtained in 5.3.2.

9.2 Separate (dry cut) the fireside half of the tube longitudinally from the shielded (or casing) half, being careful to affect the deposit as little as possible. If a band saw is used, carefully remove all fragments of metal with a magnet. Observe closely to be sure that only metal fragments are removed.

9.3 Examine the water-formed deposit (photograph if desired), and select and mark-off the boundaries of the 150-mm

(6-in.) specimen of tubing on the internal fireside that appears to be most heavily fouled and relatively uniform (undiminished by spalling). Mark a similar area on the internal casing half of the tube for comparison.

9.4 Carefully scrape the surface to dislodge and individually collect the more easily removable deposits from between the boundaries of each sample. Complete the deposit removal by brushing or applying an electric vibrating tool, or both. Dry the removed material in an oven at 105°C for 1 h. Grind sufficiently to pass through a No. 325 (45- $\mu$ m) stainless steel sieve and weigh the screened portion of each half; record the weights in milligrams.

NOTE 3—Drying the sample may affect subsequent analysis by X-ray diffraction.

NOTE 4—The purpose of the grinding and screening operation is to prevent a weighing error from chips of steel that may have been lodged in the deposit during the sample-cutting operation.

9.5 Determine the areas from which the two deposits were removed, measuring each dimension to the nearest 1 mm. Trim sheets of paper to make patterns of the actual surfaces that were stripped. If the pattern is regular in shape, determine the area by direct measurement. If the pattern is irregular, determine the area by comparing the weight of the pattern to the weight of a sheet of paper of known area.

### 10. Calculation

10.1 Determine the weight of accumulated deposits per unit area, in milligrams per square millimetre, directly by dividing the weight of deposit in milligrams by the area in square millimetres.

### 11. Precision and Bias

11.1 See 18.1.

## TEST METHOD B—SOLVENT REMOVAL

### 12. Scope

12.1 The solvent removal test method is preferable where deposition is relatively light and the deposit is adherent to the base metal.

### 13. Summary of Test Method

13.1 The deposit that has accumulated in the selected boiler tube specimen is determined by measuring the weight loss of the tube sample after deposit removal with inhibited hydrochloric acid. In the event copper plates out on the tube sample during the cleaning operation, an ammonium persulfate solution is used to remove the copper prior to final weighing. The required machining of the outside surface of the selected tube sample to a wall thickness of approximately 1 mm ( $\frac{1}{16}$  in.) will increase the sensitivity of the measurement and eliminate interference from external deposits.

### 14. Apparatus

14.1 See 8.1 through 8.5 and 8.7 through 8.10.

14.2 *Machine Lathe*, to turn down the outer surface of the sample tube.

14.3 *Hot Plate with Magnetic Stirrer*.

14.4 *Plastic Coated Stirring Bar*.

14.5 *Heat Resistant Glass or Plastic Beaker*, 1000-mL.

## 15. Reagents

15.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>4</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

15.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

15.3 *Acetone*.

15.4 *Ammonium Persulfate Solution* (10 g/L)—Dissolve 1 g of ammonium persulfate ((NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>) in 50 mL of water and add 50 mL of concentrated ammonium hydroxide (NH<sub>4</sub>OH, sp gr 0.90).

15.5 *Inhibited Hydrochloric Acid Solution* (1 + 9)—Add 100 mL of concentrated hydrochloric acid (HCl, sp gr 1.19) to 900 mL of water. Add 2 mL of appropriate inhibitor and mix well.<sup>5</sup>

15.6 *Sodium Carbonate Solution* (10 g/L)—Dissolve 10 g of sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) in 1 L of water.

## 16. Procedure

16.1 Take the 600-mm (24-in.) tube section obtained in 5.3.

16.2 Visually select the section that appears to be most heavily encrusted, assisted by suitable illumination. Select the 50-mm (2-in.) specimen from this section on which the weight of accumulated deposit per unit area will be determined.

16.3 Using the tubing cutter, isolate an approximately 150-mm (6-in.) length that will include the 50-mm (2-in.) specimen. Center the short piece in a lathe and, after marking the end with a file to show the center of the fireside, decrease the outside diameter of the tube over a length that includes the 50-mm (approximately 2-in.) specimen. When the wall thickness has been decreased to about 1.5 mm (1/16 in.), remark the fireside, if necessary, and separate this 50-mm ring or shell by applying a suitable parting tool. Then by using a milling machine (preferred) or a band saw, isolate the fireside half of this shell from the casing half and carefully remove any adhering chips. Dry the tube specimen in an oven at 105°C for 1 h.

<sup>4</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

<sup>5</sup> Dow A-120 on Rodine 213 has been found acceptable for this purpose.

16.4 Weigh the tube sample to the nearest 0.1 mg.

16.5 Remove the deposit in inhibited acid solution.

16.5.1 Immerse the specimen in a 1000-mL beaker containing 750 mL of inhibited hydrochloric acid solution (1 + 9) which has previously been brought to 70°C (160°F) on a hot plate with a plastic-coated magnetic stirrer. Keep the specimen immersed until the deposit is removed as evaluated visually. Maintain the circulation of the solvent solution and keep the solvent temperature within ±2°C throughout the test.

16.6 Remove, rinse, dry, and weigh the specimen in the following manner: Remove the specimen from the inhibited acid cleaning solution, wash in warm water, neutralize in the Na<sub>2</sub>CO<sub>3</sub> solution and immerse in the (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> solution if copper plating is present until the plating is removed. Rinse the specimen in hot water for a length of time sufficient to heat it; then dry with acetone. Weigh the dry and cleaned specimen.

NOTE 5—The specimen must be hot prior to the acetone rinse in order to prevent condensation and, hence, rusting.

16.7 Subject the clean specimen to a second 10-min treatment in the same cleaning solution, and again remove, rinse, dry, and weigh. The sample weight found should be identical within 2 mg of that recorded in 16.6, if the inhibitor is effective. If the difference is greater, it may be applied as a rough factor to correct for base metal corrosion (added to the first clean and dry weight). Base metal corrosion is generally less than 0.1 mg/cm<sup>2</sup>.

16.8 Determine the internal surface area of each specimen (use a paper pattern of the cleaned surface, if necessary) and record the area in square millimetres.

## 17. Calculation

17.1 The weight of accumulated deposit per unit area in milligrams per square millimetre is the difference in weight in milligrams of the specimen before and after acid cleaning divided by its inner surface area in square millimetres.

17.2 The weight of iron oxide removed from a “clean” boiler tube of equal area may be subtracted from the accumulated deposit weight if a more precise index of fouling is desired.

NOTE 6—A boiler tube surface coated with only a thin layer of protective oxide is generally considered “clean.”

## 18. Precision and Bias

18.1 Round-robin testing is impractical, due to the difficulty of preparing representative specimens. Precision and bias of the test method depends on the care taken during removal of the deposit as well as on the nature and uniformity of the deposit itself.

## 19. Keywords

19.1 boiler tubes; density; deposits; steam generators

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